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PATENT  
P36195 US (TH/KL)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE (EO/US)

In re Application of )

Inventor's Name: Mathias Pauls, et al. )

) Group Art: 1711

U.S. Serial No.: 09/437,276 )

) Examiner: Rachel Gorr

U.S. Filing Date: November 10, 1999 )

Int. Filing Date February 10, 1994 )

Priority Date Claimed: February 10, 1993 )

For: PREPOLYMER COMPOSITION FOR  
INSULATING FOAMS )

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Assistant Commissioner for Patents

Washington, D.C. 20231

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Howard M. Peters (Reg. No. 29,202)

DECLARATION OF MATHIAS PAULS PURSUANT TO 37 C.F.R. 1.132

Sir:

Declaration/Affidavit of Co-inventor Mathias Pauls

I, Mathias Pauls, declare that:

1. I reside at Scheregg 15, 9057 Weissbad/Switzerland, formerly at Mooshaldenstraße, 9050 Appenzell/Switzerland.
2. I am the managing director of the company to which the technology in this patent

application has been assigned to:

RATHOR AG having an address at  
Rütistrasse , 9050 Appenzell/Switzerland,

and recorded at the U.S. Patent and Trademark Office on October 16, 1995 at REEL 7900,  
frames 0916-0919.

3. I am also a named coinventor in the technology claimed in the above referenced U.S. patent application serial No. 09/437,276.
4. I have been involved in the research, technology, manufacture and marketing of polymers, polyurethanes and foams for more than 20 years. I am the inventor or coinventor of more than 12 U.S. patents or patent applications and more than 50 foreign patents or patent applications.
5. This experience and background qualify me to be considered as one having expertise in this field of technology.
6. With regard to the present invention found in U.S.Ser.No. 09/437,276, one needs to understand that the fire-retardancy is highly influenced by the polyol component involved in the prepolymer formation. It was surprisingly found that polyurethan foams formed from aromatic polyisocyanates and aromatic polyesterpolyols have better fire-retardant properties than polyurethane foams formed from aromatic polyisocyanates and polyetherpolyols, all other constituents of the foam forming compositions being the same.  
  
The considerable contribution of aromatic polyesterpolyols to the fire-retardant properties of the foams obtained from inventive prepolymer compositions is specifically addressed on page 3, lines 19 to 23 of the original specification. See also page 2, line 21 to page 3, line 6 addressing the so-called B2 requirements of European standards.
7. In comparative tests making use of aromatic polyesterpolyols vs. polyetherpolyols as polyol components in prepolymer compositions, it was shown that the B2 results of the tested foams yielded a significant better flame-retardancy for the polyesterpolyol-based foams. The B2 values are measured in mm height of flames under standardized conditions, polyesterpolyols resulting in a height of flames of 110 to 120 mm, whereas polyetherpolyols show values of 150 to 190 mm. Mixtures of polyesterpolyols and polyetherpolyols as polyol components have results in between.
8. I have attached sheets 1 and 2 describing the influence of the polyol component on the fire retardancy of the foams produced therefrom under standard conditions.
9. The corresponding EP-application no. 94 907 539.4 has been examined with the requirements of the European Patent Convention. In a hearing on July 17, 2001 and a

subsequent office action dated August 01, 2001 the European Patent Office indicated allowability of the application for prepolymers made from aromatic polyisocyanates and aromatic polyester-polyols. The required test data, see # 8 above, were filed on December 11, 2001, together with new formal documents. The communication pursuant to Rule 51 (4) EPC preceding the grant has not yet issued.

10. The to-be-allowed claims read as follows:

1. A Pressurized can for preparing polyurethane insulating foams with fire-retardant properties comprising a prepolymer composition having at least one PU-prepolymer with a content of NCO groups of 4 to 20 wt% and conventional additives, as well as a propellant component, the prepolymer component having a content of 5 to 40 wt%, based on the prepolymer component, of softening phosphates of the formula  $O = P(OR)_3$  and  $O = P(OR)_2R$ , wherein R is the same or different and selected from aryl, aralkyl and alkyl aryl groups having up to 10 carbon atoms, characterized in that the PU prepolymer is one based on aromatic polyisocyanates and polyester polyols based on ethylene glycol or glycerin and aromatic polycarboxylic acids with an OH no. of 100 to 300 and an OH functionality of 2 to 4, the prepolymer component being substantially free of halogens.
2. Pressurized can according to claim 1, characterized in that the polyisocyanate is one based on of naphthaline-1,5-diisocyanate, tolylenediisocyanate or diphenylmethanediisocyanate.
3. Pressurized can according to claim 1 or 2, characterized in that the polyester polyols have a molecular weight of 1000 to 2000.
4. Pressurized can according to one of the preceding claims, characterized by a content in liquid polybutadiene of 0.01 to 2 wt%.
5. Pressurized can according to claim 4, characterized in that the liquid polybutadiene contains about 75 % 1,4-cis double bonds, about 24 % 1,4-trans double bonds and about 1 % vinyl double bonds, has a molecular weight of about 3000 determined by vapor-pressure osmosis, and has a viscosity at 20°C of about 3000 mPa.s.
6. Pressurized can according to one of the preceding claims, characterized by a propellant content of 5 to 40 wt%.
7. Pressurized can according to one of the preceding claims, characterized in that the propellant component comprises propane, butane and/or dimethylether.
8. Pressurized can according to one of the preceding claims, characterized in that the propellant component comprises a fluorohydro carbon.
9. Pressurized can according to one of the preceding claims, characterized in that it contains

a flame-retardant additive that is free from chlorine and bromine.

10. Pressurized can according to claim 9, characterized in that the flame-retardant additive is melamine, melamine cyanurate, dimelamine phosphate, melamine phosphate, cyanodiamide, dicyanodiamide, aluminum trihydrate, ammonium polyphosphate or mixtures thereof.
11. Pressurized can according to one of the preceding claims, characterized in that the initial service viscosity of the PU prepolymer at 20°C is 5000 to 20000 mPa.s.
12. Pressurized can according to claim 11, characterized by an initial service viscosity of the PU prepolymer of 8000 to 15000 mPa.s.

Further Declarant says not.

Date: June 26., 2002

  
MATHIAS PAULS

ATTACHMENTS: PAGES 1 AND 2

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Test Report 1

Test No.	1	2	3	4	5
Desmophen PU 1578 <sup>1</sup>	380	300	200	100	0
Voranol CP 1055 <sup>2</sup>		80	180	280	380
Dislamoll DPK	543	543	543	543	543
Tegostab BF 2270	20	20	20	20	20
Tego IMR 830 (10%)	50	50	50	50	50
Texacat DMDEE	7	7	7	7	7
Polyol component, total	1000	1000	1000	1000	1000
Polyol component	275	275	275	275	275
Desmodur 44 V 20 L	385	385	385	385	385
Propellant	140	140	140	140	140
B2-values (height of flames in millimeters)	130	130	140	150	190

<sup>1</sup> polyester polyol based on phthalic acid/adipic acid, OH no. 213

<sup>2</sup> polyether polyol, OH no. 160

Test Report 2

	1	2	3	4	5	6	7	8	9	10
Desmophen PU 1578 <sup>1</sup>	380	190						240	100	
Terol 2502 <sup>2</sup>		190	380		240	140				
Stepanpol PS2402L <sup>3</sup>				380						
Desmophen PU 1708 <sup>4</sup>					140	280	380	110	220	300
Voranol P 2000 <sup>5</sup>								30	60	80
Dislamoll DPK	543	543	543	543	543	543	543	543	543	543
Tegostab BF 2270	20	20	20	20	20	20	20	20	20	20
Tego IMR 830 (10%)	50	50	50	50	50	50	50	50	50	50
Texacat DMDEE	7	7	7	7	7	7	7	7	7	7
Polyolcomponent, total	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
Polyolcomponent	287	287	287	287	287	287	287	287	287	287
Desmodur 44 V 20 L	399	399	399	399	399	399	399	399	399	399
Propellant	137	137	137	137	137	137	137	137	137	137
B2 value	110	110	110	120	120	140	150	130	140	150

<sup>1</sup> Bayer AG, polyester polyol based on phthalic acid/adipic acid, OH no. 213

<sup>2</sup> Terol 250, Oxid Europe, a recycling/polyester from PET-waste OH no. 250

<sup>3</sup> Stepanol PS 2402L, Stepan Europe, based on phthalic acid anhydride/diethylene glycol, OH no. 240

<sup>4</sup> Desmophen PU 1708, Bayer AG, polyetherpolyol, OH no. 245

<sup>5</sup> Voranol P 2000, Dow Chemical, polyetherpolyol, OH no. 50